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D E C I S I O N
of 18 May 2006

Case Number: T 0930/03 - 3.2.03

Application Number: 96306186.6

Publication Number: 0762066

IPC: F25J 3/04

Language of the proceedings: EN

Title of invention:

Production of ultra-high purity oxygen from cryogenic air separation plants

Patentee:

AIR PRODUCTS AND CHEMICALS, INC.

Opponent:

L'AIR LIQUIDE, Société Anonyme

Headword:

-

Relevant legal provisions:

EPC Art. 54, 56

Keyword:

"Novelty, inventive step: yes"

Decisions cited:

-

Catchword:

-



Case Number: T 0930/03 - 3.2.03

D E C I S I O N
of the Technical Board of Appeal 3.2.03
of 18 May 2006

Appellant:
(Opponent)

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(Patent Proprietor)

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Decision under appeal:

Decision of the Opposition Division of the
European Patent Office posted 8 July 2003
rejecting the opposition filed against European
patent No. 0762066 pursuant to Article 102(2)
EPC.

Composition of the Board:

Chairman: U. Krause
Members: Y. Jest
K. Garnett

Summary of Facts and Submissions

- I. By its decision dated 8 July 2003 the Opposition Division rejected the opposition and maintained European Patent No. 0 762 066 as granted with the following independent claims 1 and 15:

Claim 1:

"A process for the fractionation of air by cryogenic distillation using a cryogenic distillation column system comprising at least one distillation column (1,22,200) primarily separating oxygen and nitrogen from feed air (3,21,38,224), wherein a feed air stream (10) is compressed (12), cooled (20) to near its dew point and fed (3,21,221) to the distillation column system for rectification thereby producing a nitrogen-containing overhead (24) and a crude liquid oxygen bottoms (38); wherein an oxygen-containing side-draw stream (4,100,500,600) having an oxygen concentration of 1% to 35% oxygen and essentially free of heavier contaminants comprising hydrocarbons, carbon dioxide, xenon and krypton, is removed from the distillation column (1,22,200) and stripped in an auxiliary stripping column (2,102,402) to produce an ultra-high purity oxygen product (5,112,114) at the bottom of the auxiliary stripping column (2,102,402); characterized in that a portion (7,300) of liquid descending the distillation column (1,22,200) is removed from the distillation column (1,22,200) proximate to the location for withdrawing the oxygen-containing side-draw stream (4,100,500,600) for the auxiliary stripping column

(2,102,402) thereby reducing the liquid to vapor ratio in the distillation section between said location and where a top-most feed air stream (3,21,38,224) is introduced."

Claim 15:

"An apparatus for fractionation of air by a method according to Claim 1, said apparatus comprising: a cryogenic distillation column system for rectifying a compressed, cooled, feed air stream (3,21,221) to produce a nitrogen-containing overhead (24) and a crude liquid oxygen bottoms (38) and having at least one distillation column (1,22,200) primarily separating oxygen and nitrogen from feed air (3,21,38,224); an auxiliary stripping column (2,102,402) for stripping an oxygen-containing side-draw stream having an oxygen concentration of 1% to 35% oxygen and essentially free of heavier contaminants comprising hydrocarbons, carbon dioxide, xenon and krypton, to produce an ultra-high purity oxygen product; means (5,112,114) for removing said ultra-high purity oxygen product from the bottom of the auxiliary stripping column (2,102,402); and means (4,100,500,600) for conveying said oxygen-containing side-draw stream from the distillation column (1,22,200) to the auxiliary stripping column (2,102,402), characterized in that means (7,300) are provided to remove a portion of liquid descending the distillation column (1,22,200) from the distillation column (1,22,200) proximate to the location for withdrawing said oxygen-containing side-draw stream thereby reducing the liquid to vapor ratio in the distillation section between said location and where a top-most feed air stream (3,21,38,224) is introduced."

II. The Opposition Division found that the grounds of opposition, namely insufficient disclosure (Article 100b) EPC), lack of novelty and inventive step (Article 100a) EPC), and the facts submitted in these respects, did not prejudice the maintenance of the patent as granted.

III. An appeal was lodged against this decision by the Opponent on 1 September 2003; the appeal fee was paid on the same date. The statement of grounds of appeal was received on 4 November 2003.

IV. Relevant prior art considered in the appeal proceedings:

D1: EP-A- 0 446 004, corresponding to family member US-A- 5 049 173 cited in the description of the patent, paragraph [0008],

D5: US-A- 3 348 385,

D7: "Optimal Control of Distillation Systems", by Nirmal Chatterjee and S.R. Suchdeo, Air Products and Chemicals Inc, Allentown, Pennsylvania, paper presented at 1984 IETC Proceedings, conference held 15-18 April 1984, Shamrock Hilton Hotel, Houston, Texas (USA), accompanied by:

D7a: List of topics of 1984 IETC Proceedings, and

D7b: Email from Lana Tolleson, Program Coordinator Texas A&M University System, 3581 TAMU.

V. During the oral proceedings on 18 May 2006 the arguments presented by the parties were related only to the issue of inventive step.

The Opponent stated that the ground of opposition based on Article 100(b) EPC was no longer maintained in the appeal proceedings. However the objection of lack of novelty presented in the Opponent's written submissions was maintained.

VI. The Appellant (Opponent) requested that the decision of the Opposition Division rejecting the opposition be set aside and that the patent be revoked because its subject-matter lacks novelty and inventive step.

The Appellant's arguments relating to method-claim 1, which equally apply to the corresponding device-claim 15, can be summarised as follows:

(a) The method of claim 1 lacks novelty with respect to each of documents D1 and D5.

It is undisputed that the closest state of the art, D1, discloses the preamble portion of claim 1. According to the process depicted in Figures 1 and 3 of D1, a side stream (line 100) having an oxygen content comprising between 1% and 35% is withdrawn from column 22. For values near to the minimum value of 1% (see column 10, lines 6 to 11) the side-stream should be withdrawn at a region close to the top part of the column 22, thus proximate to the withdrawal of a portion of the gaseous nitrogen overhead through line 32. The nitrogen withdrawal (line 32) reduces the reflux and, therefore, the L/V ratio within the column above the point of

introduction of feed air 21. The charactering portion of claims 1 and 15 is thus also known from D1.

Document D5 discloses, in a typical double column process, the withdrawal (13) of an impure liquid nitrogen overhead from a high pressure column as product (13a) and as feed (16a) for a low pressure column. Thus, a portion (13a) of the liquid descending the column is withdrawn at the same location as the withdrawal (16) of the feed to the LP column so as to decrease the liquid to vapor ratio in that section of the column. The claimed invention is therefore likewise anticipated by D5.

(b) As to inventive step, the subject-matter of the invention lacks inventive step as shown by the following six different approaches, all of them being based on D1 as closest prior art.

- Facing the problem of providing nitrogen with conventional purity (1ppm) in gaseous and liquid form and a small amount of high purity oxygen, the skilled person would apply the recommendation of column 7, lines 18 to 20 of D1 to the apparatus of Figure 1. By doing so, nitrogen would be withdrawn from the column at a location proximate to the side withdrawal line 100 in Figure 1, which is generally provided at 1/3 of the height of the column or higher in the case of a low oxygen content of the side-draw stream in line 100. In this respect, it was emphasized that the term "proximate" is broad and that the claims do not exclude the possibility that the "section" of the column could correspond to almost the entire column.

- In the patent two problems are addressed when starting from prior art D1, namely, to control the composition of the side-draw (paragraph [0051]) and to increase the oxygen production (paragraph [0048]). The skilled person would consider the solution provided in D7, mainly as illustrated in Figure 4a at page 512, since the information contained in D7 relates to how to cope with plant disturbances affecting distillation processes, and particularly in air separation plants. D7 teaches the use of a slip-stream, which as presented in Figure 4a removes gaseous stream just below the top of the column so that the liquid to vapour ratio is increased. In turn the separation is improved and the quality, i.e. purity, of the top product increased. The skilled person would recognise the merits of such a slip-stream when applied to the product extracted from the column at the side-draw of D1, in the sense that the quality of the side-draw stream could be increased. The question as to whether the solution provided by D7 is efficient not only in short-term applications but also in a steady state is of no relevance since the claims do not comprise any limitation whatsoever in this respect.

- In the written submissions, the Appellant presented further arguments as to why it was obvious for the skilled person to provide a withdrawal of a liquid stream from the distillation column of D1 at a location close or proximate to the side-stream of oxygen product to be delivered to the stripping column.

Firstly, the passage in column 8, lines 34 to 38, of D1 suggests the enhancement of the oxygen content in the

side stream by removal of a liquid descending the column from an appropriate location. Secondly, a suggestion to remove the liquid nitrogen stream forming the reflux stream 231 (in Figure 5 of D1) from a lower section of the HP column proximate to the point of withdrawal of side stream 401 is derivable from the text in column 9, lines 5 to 8, of D1. Alternatively, the liquid side stream 100 shown in the embodiment of Figure 8 could be removed from the top of column 22 (see column 12, lines 52 to 57), thereby being proximate to the removed product streams 32,738. Finally, the embodiment of Figure 5 of D1 suggests a removal of the nitrogen product stream, together with the reflux 231 for the auxiliary stripping column (see second approach), from column 22 proximate to the point of withdrawal of the side stream 401, so as to obtain an impure liquid nitrogen product as in D5.

VII. The Respondent (Proprietor) requests dismissal of the appeal (main request) or maintenance of the patent in amended form on the basis of the claims and description submitted with letter of 17 March 2004 (first and second auxiliary requests).

The arguments presented by the Respondent can be summarised as follows:

(a) Novelty

D1, which discloses processes in accordance with the preamble of claim 1, does not disclose the removal, at a location proximate to the side-draw stream, of a portion of liquid descending the distillation column.

Referring to Figure 1 of D1, an oxygen-containing side-draw stream (100) is withdrawn from the column (22) and fed to an auxiliary stripping column (102). There is no removal of liquid descending the distillation column from a point proximate to the location for withdrawal of the side-draw stream (100) or any other arrangement to reduce the L/V between that location and the location at which the feed air stream (21) is introduced. The amount of reflux (30) lost by removal of the product portion (32) of the nitrogen-containing overhead (24) is not identical to the removed liquid portion as claimed. There is a clear distinction between descending liquid removed from a column and liquid not supplied to a column at all, and a reduction in the amount of liquid reflux (30) reduces the L/V ratio within the entire column (22) and not specifically in the section between side-draw stream withdrawal and feed air introduction.

The embodiment of Figure 3 of D1 produces UHP oxygen using a double column air separation unit, in which an oxygen-containing side-draw stream (100) is removed from an intermediate location of the HP column (22) and stripped in an auxiliary column (102) to provide gaseous UHP oxygen (112) and liquid UHP oxygen (114). There is no bypass stream that reduces the L/V in the distillation section between withdrawal of the side-draw stream (100) and introduction of the HP air feed.

D5 discloses a process of cryogenic distillation of air to provide conventional high purity (above 99%) oxygen but not UHP oxygen. A primary object of D5 is to reduce the height of an air separation plant. For that reason, a split column operation was selected and purity of the

nitrogen-containing overhead from the HP column was compromised by under-staging the HP column in order to limit its height. The disclosure of D5 does not even conform to the preamble of claim 1.

(b) The Appellant's submissions concerning lack of inventive step rely upon ex-post facto analysis and there is no reason to believe that the skilled person would have considered making any modification to the processes of D1, whether considered alone or in combination with D5 or D7 and/or common general knowledge, that would have resulted in a process of present Claim 1 or an apparatus of granted Claim 15.

None of the lines of argument offered by the Appellant demonstrated that the skilled person would have reduced the L/V ratio in a part of the distillation column by withdrawal of liquid at a location proximate to the oxygen side-draw.

- Figure 1 of D1, even in conjunction with the passage of column 7, lines 18 to 20, merely teaches the withdrawal of liquid nitrogen from the top of the distillation column. This liquid cannot be considered as descending the column or as reducing the L/V ratio in a section of the column. Even if the withdrawal of the side-draw oxygen stream 100 was located in the top third region of the column it would still not be proximate to the top where the liquid nitrogen is removed.

- Neither would the general teaching of D7 lead the skilled person to modify the process of D1 in the terms of the claimed method. The purpose of the slip-stream

disclosed in Figure 4a of D7 is to cope with temporary plant disturbances and not to provide a long-term solution for the steady-state operating mode. Furthermore, and contrary to the invention dealing with a liquid stream taken away from the liquid descending the column, Figure 4a teaches the withdrawal of a gaseous stream, followed by its condensation and reintroduction into the feed. D7 thus lacks any incentive to decrease the L/V ratio. Moreover, D7 does not suggest the location of such a withdrawal in proximity of the oxygen side-stream.

- The remaining four lines of argument presented by the Appellant also fail to demonstrate a lack of inventive step.

D1 lacks any hint or incentive to withdraw liquid descending in the LP column at a location proximate to the side-draw stream removal of oxygen. The statement at column 8, lines 34 to 38, only refers to an increased L/V ratio above the side removal of gaseous oxygen from the stripping column, rather than to a removal of liquid descending the distillation column, in the embodiment of Figure 4 of D1.

In the embodiment of D1, Figure 5, a reflux stream (231) of condensed nitrogen (26, 108) is fed from the HP column (22) to the auxiliary column. However, there is no teaching that such a stream could be withdrawn from the HP column (22) at a location proximate to withdrawal of the side-arm stream (401) fed to the auxiliary stripping column (402), especially as the reflux stream (231) for the rectifying section of the stripping column must be of significantly different

composition from the feed to the stripping section of this column.

The arrangement of Figure 8 of D1 lacks any removal of liquid descending the column proximate to any side withdrawal. Column 12, lines 52 to 57, merely indicates that a liquid nitrogen stream is withdrawn from the top of the column. This stream is not an oxygen-containing side-draw stream and no liquid descending the column is removed proximate to this stream to reduce the L/V ratio.

Similarly, D5 discloses a removal of liquid nitrogen at the top of the HP column; the skilled person is therefore not prompted by D5 to reduce the L/V ratio in a section of the column of D1.

Reasons for the Decision

1. The appeal is admissible.

2. Novelty

2.1 The invention

The Patent relates to the fractionation of air by cryogenic distillation using a cryogenic distillation column system comprising at least one distillation column primarily separating oxygen and nitrogen from feed air. As is conventional in such processes, the feed air stream is compressed, cooled to near its dew point and fed to the distillation column system for rectification, thereby producing a nitrogen-containing

overhead and crude liquid oxygen bottoms. In this type of process, a small amount of an ultra-high purity ("UHP") oxygen product containing less than 10 vppm (volume parts per million) contaminants (see [0003]) is obtained from an oxygen-containing side-draw stream removed from the distillation column and fed to an auxiliary stripping column. This side-draw stream has an oxygen concentration of 1% to 35% and is essentially free of heavier contaminants comprising hydrocarbons, carbon dioxide, xenon and krypton.

Such a process and associated apparatus are known from document D1, as acknowledged in the description of the patent see paragraph [0008].

The aim of the invention is to increase the proportion of oxygen recovered as UHP oxygen.

This is achieved by the features of the characterising portions of independent claims 1 and 15, namely by reducing the liquid to vapour ratio L/V between the location in the distillation column at which the side-draw stream is removed and the location at which feed air is introduced into the column by removing a portion of the liquid descending the column proximate to the location of withdrawal of the side-draw stream.

When assessing the teaching of this technical solution, the Board agrees with the technical interpretation given by the Opposition Division in the impugned decision.

The claimed invention requires that a portion of liquid, which is removed to reduce the L/V ratio, must descend the distillation column. Such a liquid cannot be

assimilated to liquid removed at the top of the column, which de facto cannot and does not "descend" the column. Secondly, the meaning of the term "proximate" in the context of the patent is unambiguous; it means "at or near a specific location" (see paragraph [0009]). It can in no way be interpreted as covering embodiments in which said location is substantially spaced from the location of the oxygen side-draw withdrawal.

2.2 Document D1

As acknowledged in paragraph [0008] of the patent, a process and an apparatus corresponding to the preamble portions of claims 1 and 15 is known from D1.

However the features of the characterising portions of claims 1 and 15 are not disclosed by D1.

In particular, there is no explicit or implicit teaching in D1 to reduce the L/V ratio in the column between the location of the oxygen side-draw stream and the feed air.

In the process depicted in Figures 1 and 3 of D1 a portion of the overhead gaseous nitrogen is withdrawn from the distillation column 22 and removed as a gaseous nitrogen product via lines 32,34. The removal of this stream indeed reduces the reflux and thereby the L/V ratio in the distillation column. However, this applies to the whole column, rather than to the section between the locations of the withdrawal of side stream 100 and the feed 21, and is obtained by removing the gaseous nitrogen stream at the top of the column rather than liquid descending the column.

On the other hand, the oxygen-enriched liquid side stream is withdrawn at the side of column 22 via line 100 of Figure 1, thus not at the top thereof. If there had been any intention in D1 that the relevant stream could have been drawn from the top of the column, the qualification that the stream is a side-draw stream would not have been present. In this connection, side-draw clearly does not mean simply withdrawn from the side of the column even if the location is at the top of the column because the direction of withdrawal of a stream makes no difference to its composition. Further, since the stream is to be fed to an auxiliary stripping column to provide UHP oxygen, there would be no reason to withdraw the stream from a location (the top of the column) at which the oxygen concentration in the column is at its lowest and well above the level in the column at which there are essentially no heavier contaminants. Even if the oxygen content of the side-stream was chosen to be as low as 1%, which is the lower limit specified in column 10, lines 6 to 11, the location of the side-draw stream would be considerably below the top of the column 22, where nitrogen is removed as a product and where, therefore, the oxygen concentration is substantially below 1%. As a consequence, the "removal" of the liquid in the column by reducing the reflux at the top of the column would not be proximate to the location for withdrawing the side-draw stream even if the oxygen concentration thereof was as low as 1%.

Finally, it appears that Figure 5 in the patent cannot support a broad interpretation of the word "proximate" as including such differing points of withdrawal because the text on page 6, lines 45/46, makes clear

that the line 300 should be at the same location as the withdrawal of the side stream through line 500 so that the drawn reference line 300 is obviously mistaken.

2.3 Document D5

Whereas document D5 discloses, in a typical double column process, the withdrawal (13) of an impure liquid nitrogen overhead from a high pressure column as product (13a) and feed (16a) for a low pressure column, the withdrawn streams neither form a "side-draw stream" nor remove a portion of liquid "descending" the high pressure column to thereby reduce the L/V ratio in a specific section of the column according to the clear meaning of the terms utilised in claim 1. Further, the stream 16a is fed to a typical low pressure column rather than to a stripping column for producing UHP oxygen.

2.4 The requirement of novelty (Article 54(1) EPC) is therefore fulfilled.

3. Inventive step

3.1 Closest prior art - Technical problem

As mentioned above, D1 is indisputably the closest prior art and discloses the process and apparatus as defined in the respective preamble portion of independent claims 1 and 15.

The difference between the invention and the closest prior art can be summarised and defined as the reduction of the L/V ratio in the column between the

location of the oxygen side-draw stream and the feed air by removing a portion of liquid descending the air distillation column. In doing so, the oxygen concentration of the oxygen-containing side-draw stream is increased while still ensuring that the oxygen-containing side-draw stream is free of heavies.

The technical problem solved by the invention is to improve the oxygen production from the stripping column without sacrificing oxygen purity.

3.2 The Appellant submitted six different approaches to demonstrate a lack of inventive step, all of them being based on D1, and one of them relying on the combination of D1 and D7.

3.2.1 The mode of realisation of Figure 1 of D1 merely teaches the withdrawal of gaseous nitrogen from the top of the distillation column. This stream cannot be considered as being a liquid descending the column or as reducing the L/V ratio in a section of the column. But even if additional liquid nitrogen was withdrawn as a fraction of the condensed nitrogen stream 30, as could be derived from the description at column 7, lines 18 to 20, this would take place at the top of the column rather than proximate to the side-draw oxygen stream 100. The skilled person would indeed not "move upwards" the side-draw 100 for the oxygen to be delivered to the stripping column to the top region where the oxygen content is far below 1% because it is well-established that such an oxygen stream is taken from the lower part of the distillation column, namely at a level where, and as soon as, the heavies

originally present in the stream have essentially been removed.

- 3.2.2 Neither would the general teaching of D7 which, in view of D7a, can be acknowledged as prior art because made available to the public in Summer 1984, lead the skilled person to modify the process of D1 in the terms of the claimed method. The purpose of the slip-stream disclosed in Figure 4a of D7 is to cope with temporary plant disturbances and not to provide a long-term solution for the steady-state operating mode. Furthermore, and contrary to the invention dealing with a liquid stream taken away from the liquid descending the column, Figure 4a teaches the withdrawal of a gaseous stream, followed by its condensation and reintroduction into the feed. This does not affect the L/V ratio below the location of withdrawal of the slip stream and therefore no incentive to decrease the L/V ratio can be derived from D7. Moreover, D7 does not suggest the location of such a withdrawal in proximity to an oxygen side-stream, which does not exist in D7.
- 3.2.3 None of the remaining third to sixth lines of argument as submitted in writing by the Appellant persuades the Board that it would have been obvious for the skilled person to amend the state of the art disclosed in D1 by:
- removing a portion of liquid descending the distillation column from the distillation column proximate to the location for withdrawing the oxygen-containing side-draw stream for the auxiliary stripping column so as to reduce the L/V ratio in the distillation section between said location and where a top-most feed air stream is introduced.

- The information contained in column 8, lines 34 to 38, of D1 does not suggest the enhancement of the oxygen content in the side stream by removal of liquid descending the column from an appropriate location. First, the side stream 360 is removed from the stripping column rather than from the distillation column and cannot, therefore, affect the oxygen content in the feed to the stripping column. Second, the removed stream 360 is gaseous (normal purity gaseous oxygen, see column 8, lines 34 to 38), rather than liquid, and it is said to "decrease" the L/V ratio "in this section", which should be construed as meaning that the L/V ratio in the lower section is lower than the ratio in the upper section of the column due to the removal of gaseous stream 360 between these sections.

- The text in column 9, lines 5 to 8, of D1 does not suggest the removal of the liquid nitrogen stream forming the reflux stream 231 (in Figure 5 of D1) from a location of the HP column proximate to the point of withdrawal of side stream 401. It should be noted that the introduction of the reflux stream 231 into the auxiliary stripping column 402 above the introduction of feed stream 407 implies a difference in composition of both streams, further implying a significant difference in the levels between the corresponding points of withdrawal from column 22, and therefore excluding any "proximate" location of both points.

- In the fifth approach, it is argued that, in the embodiment of Figure 8, the liquid side stream 100 could be removed from the top of column 22 (see column 12, lines 52 to 57), thereby being proximate to

the removed product stream 32. Even if the cited passage of D1 could be read as suggesting the removal of the stream 100 as a side-draw stream somewhat below the top of column 12, the nitrogen product would still have to be withdrawn in gaseous form from the top of the column, i.e. at a different level, and this would then not be consistent with the removal of a portion of liquid descending the column for reducing the L/V ratio in a specific section of the column.

- Finally, the skilled person would not have modified the embodiment of Figure 5 of D1 by the removal of the nitrogen product stream, together with the reflux 231 for the auxiliary stripping column, from column 22 at a location proximate to the point of withdrawal of the side stream 401, even if he had taken D5 into account. Apart from the fact that D5 proposes neither the withdrawal of an oxygen containing stream as a side-draw stream from the HP column nor the removal of liquid descending this column, the incompatibility of the proposed modification with the generally required concentration profile within the auxiliary stripping column would have prevented the skilled person from removing the reflux stream for the stripping column from the HP column at a location proximate to that of stream 401.

3.3 The process of claim 1 as well as the apparatus of claim 15 thus involves an inventive step in the meaning of Article 56 EPC.

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar:

The Chairman:

A. Counillon

U. Krause